MANUFACTURE OF STRONG, LIGHTWEIGHT CARRIER GRANULES

FIELD OF THE INVENTION

[0001] This invention relates to the manufacture of carrier granules suitable for use as carriers for herbicides, insecticides, and fungicides, for plant growth regulators, and for other biologically active compounds. The carrier granules of this invention are made of a mix of wood fibers, powdered minerals, and binder. The small particle size of these ingredients optimize their mixing within the granule, creating a homogenous structure. The unique properties of the fibrous component herein and the innovative manufacturing method of this invention provides carrier granules that have a superior balance between strength, size, and weight.

BACKGROUND OF THE INVENTION

[0002] Manufacture granular particles are conventionally used as carriers for agricultural chemicals such as herbicides, plant growth regulators, fertilizers, etc. Various different types of granules are currently used for such applications. Patents disclosing granules include the following.

[0003] US 5,019,564 discloses granules formed by the agitated agglomeration of slurries containing plant fibers and mineral fillers. These granules are utilized as carriers for biologically active chemical agents.

[0004] US 5,078,779 discloses binder compositions that include reactive carbonates and reactive sulfates in combination with silicate

strengthening agents and water-dispersing agents. These binders are used to carry ammonium sulfate fertilizers.

[0005] US 5,242,690 discloses granular carrier compositions that include grain dust and a binder of calcium or sodium lignosulfonate, the compositions being useful as carriers for biologically active chemical agents.

[0006] US 6,613,138 discloses granules that include one or more mineral components having a bulk density greater than about 70 pounds per cubic foot, one or more lightweight additives, and one or more water soluble binders. The granules are useful as carriers for herbicides, plant growth regulators, insecticides, fungicides, or other pesticides. The patent teaches that the lightweight additives should be a non-fibrous material, because fibrous material can adversely impact the dispersibility and the flow characteristics of the finished granular substrate.

[0007] There is, however, an unmet need for carrier granules that will better satisfy the needs of industry. In particular, there is a need for small, light granules, having particles in the size range 10/40 Mesh and weighing approximately, for instance, around 28 pounds per cubic foot. The carrier granules should also have a Resistance to Attrition rating of greater than 95%. While some of these characteristics are found in some currently available granules, we are unaware of any carrier granules that provide all of these characteristics at the same time. A purpose of this invention is to provide carrier granules having particles in the size range 10/40 Mesh and weighing on the order of magnitude of 28 pounds per cubic foot and having a Resistance to Attrition rating higher than 95%.

SUMMARY OF THE INVENTION

[0008] The present invention provides a novel method for the manufacture of improved carrier granules. The method of this invention forms a mixture comprising (A) fibers, (B) mineral filler, and (C) binder. The mixture contains 32-45 weight-% cellulose fibers having a bulk density of less than 20 pounds per cubic foot, at least 35 weight-% of which fibers are retained on a 50-Mesh U.S. Sieve Series screen. The fibers employed in this invention generally have a moisture content of less than 15 weight-%. The mixture also contains 53-65 weight-% mineral filler having a moisture content of less than 12 weight-% and a bulk density of less than 65 pounds per cubic foot. In addition, the mixture contains 3-7 weight-% binder. The method of this invention agglomerates the mixture by conditioning and agglomerating it in a pin mixer to form small particles followed by agglomerating the mixture in a disc or pan pelletizer to form substantially spherical granules. The method of this invention then dries the granules, generally to a moisture content of less than about 5 weight-%, and screens the granules, to select granules that pass through a 10-Mesh U.S. Sieve Series screen but are retained on a 40-Mesh U.S. Sieve Series screen.

[0009] A carrier granule embodiment of the present invention contains: 32-45 weight-%, more preferably 35-40 weight-%, of cellulose fibers having a bulk density of less than 20 pounds per cubic foot, wherein at least 35 weight-% of said fibers are retained on a 50-Mesh U.S. Sieve Series screen; 53-65 weight-%, more preferably 54-60 weight-%, mineral filler having a bulk density of less than 65 pounds per cubic foot; and 3-7 weight-%, more preferably 5-6 weight-%, binder. The mineral filler is preferably kaolin, titanium dioxide, sodium bicarbonate, calcium carbonate, lime, fly ash, dolomite, gypsum, or mixtures thereof, and preferably has a particle size range within the range 10 to 500 microns. Calcium carbonate (bulk density

about 60 lbs/ft^3) is particularly preferred as a mineral filler. The binder is preferably granules of superabsorbent polymer, water-soluble starch, acrylic polymer, polyvinyl acetate, guar gum, or mixtures thereof, and at least 70% of the binder granules preferably are sized to pass through a 200-Mesh U.S. Sieve Series screen. Unmodified starch is particularly preferred as a binder.

[0010] In accordance with this invention, the carrier granule passes through a 10-Mesh U.S. Sieve Series screen and is retained on a 40-Mesh U.S. Sieve Series screen. The granule has a moisture content of less than 5 weight-%. The granule has a resistance to attrition of at least 95%. And the granule has a bulk density of less than 40 pounds per cubic foot, preferably 25-30 pounds per cubic foot.

[0011] The carrier granules of this invention may be used, for instance, in pesticidal compositions. Such pesticidal compositions will include a pesticidally effective amount of a pesticide releasably carried on a carrier granule as described above.

BRIEF DESCRIPTION OF THE DRAWINGS

[0012] Figure 1A shows a combination of long and short fibers for use in the present invention.

[0013] Figure 1B shows Medium-Density Fiberboard (MDF) fibers.

[0014] Figure 2A shows a carrier granule of the present invention.

[0015] Figure 2B shows a granule made with MDF fibers.

[0016] Figure 2C shows a paper sludge granule.

DESCRIPTION OF PREFERRED EMBODIMENTS

[0017] In the present invention, the wood (cellulose) fibers serve as a structural skeleton for the granules. They also contribute to the formation of cavities (pores) within the granules, thus reducing product weight and improving the release of active chemicals (pesticides, fertilizers, etc.). The fiber size distribution of the cellulose fibers in the granules will preferably provide a combination of short and long fibers that will contribute to the development of a strong yet open structure in the granules. The dry cellulose fibers in the granule will generally have a length of up to 2 millimeters, with a minimum of 35% of the fibers being retained on a 50-Mesh U.S. Sieve Series screen. For a tabulation of U.S. Sieve Series screen nomenclature, see Perry's Chemical Engineering Handbook, 6th Ed., McGraw-Hill, Inc., New York, N.Y. (1984), p. 21-15 (Table 21-6). Inasmuch as the median granule size in this invention is approximately 20-Mesh, which = 0.841 mm, such fibers are – in the context of this invention and compared to flour-like fibers – "long". The cellulose fibers preferably have a moisture content of less than 15 weight-%. Long "fluffy" wood fibers which weigh less than 20 pounds per cubic foot are preferred.

[0018] Cellulose fibers usable in accordance with this invention may be made from sawdust and similar waste or by-product of hardwood and softwood manufacturing facilities. Fibers in the wood waste as received vary in length, e.g. from 15 mm to 10 microns. The required size fibers for this invention (from about 10 microns to about 2 mm) are obtained by passing the wood through a hammer mill and employing screening to select out fibers of the desired sizes.

[0019] Figures 1A and 1B are photographs of two different types of wood fibers. Figure 1A shows a combination of long and short fibers that can be used in the present invention. The long fibers in Figure 1A will be retained on a 50-Mesh U.S. Sieve Series screen. Figure 1B shows Medium-Density Fiberboard (MDF) fibers. These fibers are used in the lumber industry to make high grade plywood-type products; they usually contain urea formaldehyde binder. The MDF fibers are very small, in fact, flour-like. They are not suitable for practicing the preferred embodiments of the present invention.

[0020] The mineral in the granule is a filler which gives the granule its desired specific weight. Fillers that may be used include kaolin, titanium dioxide, sodium bicarbonate, calcium carbonate, and mixtures thereof. In a preferred embodiment of the invention, this filler is a lime derivative, e.g., lime itself, fly ash, dolomite, calcium carbonate, gypsum, and mixtures thereof. However, any inert, preferably low pH mineral that has a high specific weight and is capable of supplying fine particles may be. Calcium carbonate or agricultural lime is currently preferred. Generally, the dry mineral filler has a particle size range within the range 10 to 500 microns, and has a moisture content of less than 12 weight-%. Any mineral filler with particles smaller than 30-Mesh U.S. Sieve Series will be operative in the present invention. In accordance with the present invention, the mineral filler has a bulk density of less than 65 pounds per cubic foot.

[0021] The binder assists the cellulose fibers in providing structural form to the granules. One or more than one binder material may be used. Binders may be selected from amongst organic binders, synthetic binders, and polymeric binders including superabsorbent polymers. Typical binders that may be used in this invention include starch, acrylic polymer, polyvinyl acetate, guar gum, and mixtures thereof. It is currently preferred to employ

a starch that dissolves well in cold water as the binder. More preferably, the binder is constituted of unmodified starch granules, at least 70% of which pass through a 200-Mesh U.S. Sieve Series screen.

MANUFACTURE

[0022] To manufacture the granules of this invention, a homogenous granule mixture is prepared and then is pelletized and the pellets are dried and screened. More specifically, this invention contemplates a method for making a granule, which method includes the steps of: forming a mixture comprising 32-45 weight-% dry cellulose fibers, 55-60 weight-% dry mineral filler, and 3-7 weight-% binder; pelletizing the mixture in a pin mixer and disc or pan pelletizer to form substantially spherical granules; and screening the granules to select granules which, for instance, pass a 10-Mesh U.S. Sieve Series screen but are retained on a 40-Mesh U.S. Sieve Series screen.

[0023] *Dry Blends Preparation.* In this stage, a bulk mixture of components in the desired weight ratios is prepared. Each scheduled component is dosed in its turn from a weighing station into a hopper. Once all of the components are in the hopper, the unmixed batch is conveyed to a mixer. The components, which at this point differ in bulk density and texture, require intensive mixing to achieve a good mix. A typical mixing procedure mixes each batch for from 90 to 120 seconds in a plowshare high-speed mixer. Once well mixed, each batch is conveyed to a surge and combined with other batches having the same component weight ratios.

[0024] *Agglomeration.* This stage creates granules from a dry blend of granule components. Dry blend is dosed continuously into a pin mixer. At the same time, water is injected into the pin mixer at several different locations. High-speed rotation of the wetted blend within the pin mixer

creates "seeds" or small particles of the blended materials. During this step, the cellulose fibers are "conditioned" or softened by the water. This conditioning step is important in the present invention due to our use of long fibers. The wetted blend is then transferred to an agglomeration pan, where agglomeration is completed. In the agglomeration pan, more material accumulates around each seed, and more waters is added, creating a more spherical granule. Parameters such as granule size and weight can be controlled in this stage by changing the blend/water ratio as well as by changing the speed and/or inclination of the pin mixer and/or the agglomeration pan.

[0025] *Compacting.* At this point, the wet granules are compacted in a rotating drum. This imparts their final strength and bulk density characteristics. Variations in strength and/or bulk density can be achieved by adjusting the length of time and/or speed of rotation, following empirical determination of relationships between rotation and those characteristics.

[0026] *Drying.* In this stage, wet granules are dried to reach their final moisture level. The open structure of the granules reduces significantly the drying time and the energy required for drying. Perforated belt dryers or fluidized bed dryers are employed to remove the necessary amount of moisture from each granule. The desired final moisture content, generally from about 2% to about 4%, is achieved by controlling the air temperature, air speed, and granule throughput rate in the dryer.

[0027] *Dry Screening.* Once dried, the batch of granules of this invention may be screened to remove both oversized granules and undersized granules, and to provide a product having a uniform granule size profile. Those skilled in the art are familiar with appropriate screening technology and the use of such devices as vibrating and rolling machines.

The oversized and undersized granules are recycled to the Dry Blend Preparation stage.

[0028] GRANULES. Figure 2A shows a granule manufactured in accordance with the present invention. The carrier granule of this invention is coherent in shape, while providing a somewhat open and porous structure. Long fibers can be seen at the surface of this granule. In contrast, Figure 2B shows a granule made with MDF fibers. Figure 2C shows a paper sludge granule. The granules of Figures 2B and 2C have a dense appearance. They lack openness and porosity of the present invention and are significantly heavier.

[0029] SPECIFIC FORMULATIONS. Typical specific formulations are set forth below. Those skilled in the art will recognize that the specific ingredients recited and their relative amounts can be varied widely while still making available the benefits provided by the present invention.

[0030] Example # 1

Components	**Weight-%**
Wood Fibers	40%
Calcium Carbonate	54%
Unmodified Starch	6%
Total	100%

[0031] Example # 2

Components	**Weight-%**
Wood Fibers	35%
Calcium Carbonate	60%
Unmodified Starch	5%
Total	100%

[0032] Examples # 3 and # 4

In these Examples, various test procedures are used to determine significant properties of the carrier granules. The carrier granules tested are made of hardwood fibers, calcium carbonate, and binder. They differ with respect to their granule size profiles. ASTM E727-02 is entitled "Test Methods for Determining Bulk Density of Granular Carriers and Granular Pesticides". ASTM E728-91 is entitled "Standard Test Method for Resistance to Attrition of Granular Carriers and Granular Pesticides". ASTM E1521-98 is entitled "Test Method for Liquid Holding Capacity of Granular Carriers". ISO 8398-89 is entitled "Solid Fertilizers – Measurement of Static Angle of Repose". The procedures for carrying out these standard tests are expressly incorporated by reference herein.

14/40 Mesh		
Bulk density	ASTM E727-01	27.5 lbs/ft^3
Resistance to attrition	ASTM E728-91	95%
Liquid holding capacity	ASTM E1521-98	25%
Angle of repose	ISO 8398-89	39.0°
Moisture content		1-2%
pH		7-8
Visual dust*		2-3 seconds

10/14 Mesh		
Bulk density	ASTM E727-01	28.0 lbs/ft^3
Resistance to attrition	ASTM E728-91	98%
Liquid holding capacity	ASTM E1521-98	27%
Angle of repose	ISO 8398-89	39.5°
Moisture content		1-2%
pH		7-8
Visual dust*		2-3 seconds

* The "visual dust" determination is a visual determination of airborne dust. The time given is the time it takes for virtually all of the dust to dissipate in the following procedure. 3 liters of product are placed in a tray and poured from a height of 14 inches into another tray, at a constant flow rate that pours out all of the product in 6-8 seconds. Once the sample has been completely poured out, a stopwatch is activated. When all of the dust has settled, the stopwatch is stopped and the time necessary for all of the dust to settle. This measurement, of the number of seconds required for the dust to dissipate, indicated the relative dustiness of the sample.

[0033] Example # 5: USE AS A CARRIER

100 pounds of granules of Example 1 and 5 pounds of carbaryl (Sevin) pesticide dispersed in 10 gallons of water are dosed in turn from a weighing station into a hopper. The unmixed batch is conveyed to a plowshare high-speed mixer, where they are mixed for from 90 to 120 seconds in a plowshare high-speed mixer to provide a product that can be used to deliver the carbaryl pesticide to lawns.